ments. (2) Using the method outlined in our paper it is a simple matter to evaluate the expressions for the diamagnetic susceptibility of holes in germanium and silicon, using Wilson's[30] equation (6.8.1):

$$\chi=\frac{e^2}{12\pi h^2c^2}\int\left[\frac{\partial^2\epsilon}{\partial k_x^2}\frac{\partial^2\epsilon}{\partial k_y^2}-\left(\frac{\partial^2\epsilon}{\partial k_x\partial k_y}\right)^2\right]\frac{\partial f_0}{\partial\epsilon}dV_k.$$

[30] See reference 6, p. 175.

If we apply the expansion scheme in spherical coordinates we obtain

$$\chi=\frac{1}{6}\frac{e^2}{hc^2}\left(\frac{KT}{2\pi m_0}\right)\exp[(\epsilon_v-\epsilon_F)/KT](A\pm B')^{\frac{1}{2}}\{\gamma_\chi\},$$

where $\{\gamma_\chi\}$ is the γ series which appears above in the expression for σ_{ijl}, Eq. (12).

PHYSICAL REVIEW VOLUME 100, NUMBER 6 DECEMBER 15, 1955

Mechanical Strength of Thin Films of Metals*

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(Received September 12, 1955)

The tensile strength of silver films are determined as a function of their thickness. The films are electrodeposited on the complete cylindrical surfaces of small steel rotors and the rotor speeds necessary to throw them off are determined. Both the tensile strength and the adhesion of the films are obtained by using rotors of different radii. Also, the adhesion is measured by electrodepositing the films on the rotor in circumferentially disconnected patches which eliminate the hoop stresses. In most of the measurements, the adhesion is made vanishingly small by dipping the rotors in an albumin solution before electrodepositing the film and by thermal cycling. The tensile strengths of the films thicker than about 6×10^{-5} cm are found to be independent of thickness and approximately equal to that of bulk silver at the corresponding temperature. For thickness from 6×10^{-5} cm to 2.5×10^{-5} cm the data show some scatter, but below 2.5×10^{-5} cm the tensile strength increases many-fold. The region where the tensile strength increases very rapidly is slightly dependent upon the electrodeposition current but within experimental error is independent of film temperature.

UNDER the influence of tensile or shearing forces, a metal deforms elastically at first, and then plastically. Usually, plastic flow begins with stresses at least 100 times smaller than should be required to cause whole atomic planes in a perfect metal crystal to move over one another. This discrepancy between the caculated and observed values of the stresses required to produce plastic flow is generally attributed to the movement of lattice imperfections or dislocations which are believed to exist in most ordinary metal crystals.[1] This dislocation theory has been remarkably successful, not only in accounting for the low mechanical strength of metal crystals, but also for many other observed properties of substance that otherwise would be difficult to understand.[2] Consequently, if it were possible to free a metal crystal of its dislocations or to lock them so that they could not move, the stresses required to produce plastic flow would be increased enormously. Furthermore, in polycrystalline metals, where at temperatures well below their melting points fracture generally is observed to be transcrystalline,[3,4] increased strength should also be obtained. Apparently, the above conditions are at least partially realized experimentally, by forming the material in sufficiently fine filaments or thin sheets. Griffith[5] found that under certain conditions the tensile strengths of very fine fibers of glass and of metals, were many times that of the bulk material from which they were made, while Orowan[6] observed that extremely thin sheets of mica were very strong. More recently, Herring and Galt[7] and others, have found that metal crystals (whiskers), approximately 18×10^{-5} cm in diameter, are several orders of magnitude stronger than the ordinary bulk metal. In some preliminary experiments[8] an increase in the tensile strength of thin films of silver has been observed when their thickness was reduced to between 10^{-4} cm and 10^{-5} cm. The purpose of this paper is to report, in more detail, an extension and improvement of these measurements.

In order to prevent tearing or stress concentrations in the very thin films, a centrifugal field has been used to apply the stresses. The method consists in uniformly depositing thin metal films on the cylindrical surface of a small rotor with rounded ends and then spinning the rotor in a vacuum until the film is thrown off. By this procedure, accurately known stresses may be applied

* Supported by the Office of Ordnance Research.
[1] A. H. Cottrell, *Progress in Metal Physics* (Interscience Publishers, New York, 1949), Vol. 1, p. 770.
[2] Seitz, Shockley, Read, and Mott in *Imperfections in Nearly Perfect Crystals* (John Wiley and Sons, Inc., New York, 1952).
[3] R. King and B. Chalmers, *Progress in Metal Physics* (Interscience Publishers, New York, 1949), Vol. 1, p. 127.
[4] C. S. Barrett, *Structure of Metals* (McGraw-Hill Book Company, Inc., New York, 1952), second edition.
[5] A. A. Griffith, Trans. Roy. Soc. (London) **221**, 163 (1921).
[6] E. Orowan, Z. Physik **82**, 235 (1933).
[7] C. Herring and J. K. Galt, Phys. Rev. **85**, 1060 (1952).
[8] Beams, Walker, and Morton, Phys. Rev. **87**, 524 (1952). Beams, Morton, and Turner, Science **118**, 567 (1953).

uniformly, and at almost any desired rate. By assuming that the centrifugal action is opposed by the hoop stresses, and the adhesion of the film to the rotor, it can be shown that in the case of a hollow thin-walled cylindrical rotor, unsupported at the ends,

$$4\pi^2N^2R^2d = T + AR/h, \tag{1}$$

where N is the rotor speed in rps, R is the rotor radius, d the density of the deposited film, h the thin-film thickness, T the tensile strength of the film, and A the adhesion.

It will be observed that the second term on the right contains R/h, while the first does not, so that by experimenting with rotors of different radii, both the tensile strength and the adhesion can be obtained. On the other hand, if the ratio of the rotor radius to the film thickness is large, the second term on the right of Eq. (1) overshadows the first, and, unless the adhesion is small, minute variations in the adhesion make it difficult to get reliable values for the tensile strength. Although experiments are underway in which it is hoped that this ratio can be greatly reduced, in the present experiments this ratio is several hundred for the thinnest films. Consequently, the films have been deposited and treated in such a way that the adhesion is small.

If the adhesion of the film is vanishingly small, $4\pi^2N^2R^2d = T$, which shows that the tensile strength is independent of the film thickness. This neglects the small effect of the circumferential strain or stretching of the film, and assumes that the film is thin and behaves plastically in a homogeneous way. On the other hand, if the hoop stress vanishes, as in the case when the material is deposited on the rotor surface in circumferentially disconnected patches, then the adhesion is given by $A = 4\pi^2N^2Rhd$.

DESCRIPTION OF EXPERIMENTAL ARRANGEMENT

The rotors are suspended magnetically in a vacuum and are spun around a vertical axis by a rotating magnetic field. The method is a modification of one previously described.[9] Figure 1 shows a schematic diagram of a magnetic suspension apparatus for spinning rotors at various temperatures. The steel rotor is freely suspended inside of a glass vacuum chamber by the axially-symmetric diverging magnetic field of the solenoid. The vacuum chamber is surrounded by two concentric glass Dewar vessels, and the solenoid which is outside the outer Dewar vessel. They all have a common axis, as shown in Fig. 1. A small pickup coil, mounted inside the inner Dewar, and just below the vacuum chamber, serves as a sensing element of a servo-circuit, which regulates the current through the solenoid in such a way that the rotor is maintained at the desired vertical position. The rotor automatically seeks the strongest part of the field which is on the axis of the solenoid. However, if disturbed, it swings around the axis. In order to prevent these horizontal oscillations, a steel damping needle (1 cm long and 0.1 cm diameter) is placed just below the sensing coil, and fastened to a weight in the inner Dewar with a fine gold chain 3.5 cm long. In order to spin the rotor at reduced temperatures, the two Dewars are filled with various liquids, i.e., when liquid nitrogen temperature is desired, they are filled with liquid nitrogen, etc. If the rotor starts to swing, the magnetic field follows it, and hence pulls the needle along with it. The viscous drag produced by the liquid on the damper is sufficient to stop the horizontal motion of the rotor. A cone-shaped plastic shield around the damper keeps bubbles formed near the bottom of the inner Dewar from disturbing the damper. The support solenoid consists of 21 110 turns of No. 23 *AGW* enamel copper wire wound on a plastic form. It has a resistance of 710 ohms, an inductance of 37 henries, and a disturbed capacity of 380 $\mu\mu$f. The support circuit is shown in Fig. 2. The pick up coil L_1 is the inductive element in the grid circuit of a partially neutralized tuned-grid, tuned-plate oscillator which operates at about 6 Mc/sec. A change in the vertical

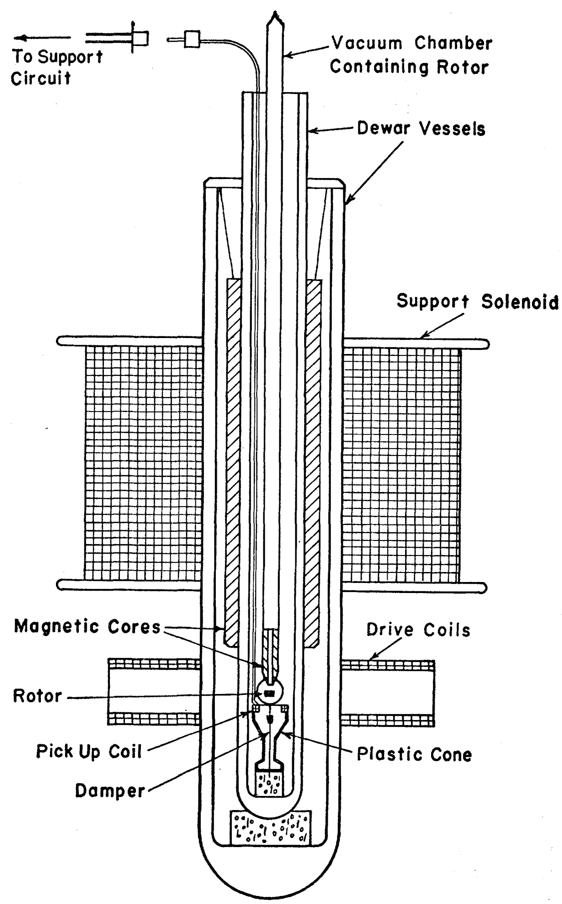

FIG. 1. Diagram of appratus for spinning rotors at various temperatures.

[9] Beams, Young, and Moore, J. Appl. Phys. 17, 886 (1946).

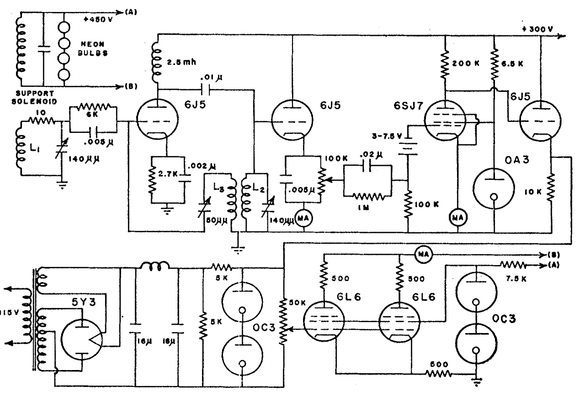

FIG. 2. Magnetic suspension circuit.

position of the rotor with respect to the pickup coil changes the Q of the oscillator circuit, and hence the amplitude of the oscillations. The output of the oscillator is applied to the grid of a 6J5 in an infinite-impedance detector stage which does not load the oscillator. The dc voltage appearing across the cathode resistor is proportional to the amplitude of the oscillations, and therefore is an electrical measure of the rotor height above the pickup coil. This output of the detector is amplified and used to control the grid voltage, and thus the plate current of the power amplifier, which is also the current through the support solenoid. The result is that any change in the position of the rotor produces a change in the support solenoid current which tends to correct the original change in position. In order to prevent vertical hunting of the rotor, a phase lead, together with effective damping is introduced into the amplifier stage, which prevents all observable vertical hunting. If desired, the current in the solenoid required to support the rotor may be reduced by placing a soft iron core inside the vacuum chamber, together with a soft iron sleeve inside the outer Dewar as shown in Fig. 1.

Two shapes of rotors were used. The first consisted of a solid cylinder 0.55 cm in diameter, and 0.20 cm high, on top of which was an inverted cone 0.318 cm high, and with half-angle of 30°. The second type was a solid cylinder 0.276 cm in diameter, and 0.234 cm high (the length of the cylinder is less than $\sqrt{3}$ times its radius). The rotors were made from 0.95/1.10 carbon tool steel, which was heated to 1450°F and quenched in water. They were tempered by heating to 425°F and quenching in water. They were then polished with emory paper, starting with No. 0 and progressing down to No. 0000. Some of the rotors were polished further with jewelers' rouge. In some of the low-temperature experiments, a special steel was used for the rotors, with coefficients of expansion somewhat closer to that of silver. The silver films were deposited on the rotors by electro-deposition. Contrary to most commercial electroplating problems, where good film adherence is of importance, the film adherence was purposely reduced to a minimum. The silver was deposited from a fresh cyanide solution containing 31 gm/l of silver cyanide, 45 g/l of potassium cyanide and 55 gm/l of potassium carbonate, maintained at 32.4=0.1°C. The rotors to be plated were carefully cleaned in acetone, then in warm water containing a commercial detergent (Surf), rinsed thoroughly, and dried. They were then dipped in a solution of 0.2 g/l of purified human albumin in water, as recommended by Jacquet[10] for eliminating adhesion. The thin layer of albumin which remains on the rotor is conducting, and hence allows the electrodeposition of excellent films, but reduces their adhesion to a negligible value. In addition, some of the rotors were thermally cycled by dipping in liquid nitrogen, to further reduce adhesion. The rotor was spun at 2 rpm in the center of the anode which consisted of a helix of 1 mm pure silver wire during the electrodeposition. When patches were plated on the rotor, thin sections of plastic were fitted over portions of the rotor which were to be left bare. The rotor is spun inside the vacuum chamber by a rotating magnetic field in a manner similar to that of the armature of an induction motor. The method has been described in detail previously.[9] During the accelerating period, the rotor is, of course, heated by the eddy currents induced by the rotating magnetic field, but by using very slow acceleration rates, especially at speeds near where the film ruptures, the heating was made comparatively small. However, especially at temperatures below that of liquid nitrogen, this heating is greater than can be tolerated, so we are developing a method in which the rotor accelerates as a synchronous motor in which the frequency of the rotating magnetic field increases at the same rate as the rotor speed. This reduces the eddy currents and the heating to a negligible value. The rotor friction due to the mangetic suspension, is extremely small, and can be accounted for entirely as due to gaseous friction on the spinning rotor. By maintaining the gas pressure in the chamber surrounding the rotor below 10^{-6} mm of Hg this friction is very minute, and hence produces no appreciable heating. In order to stop the rotor in a reasonable time, the direction of rotation of the magnetic field must be reversed and the rotor driven to rest. The rotor speed is determined by reflecting light off the rotor into a photomultiplier tube. A mark on the top (or upper cone) of the rotor causes the output of the photomultiplier tube to be periodic, with a repetition rate equal to the speed of the rotor. This output signal is amplified and compared with that from a variable-frequency oscillator on a cathode-ray oscilloscope screen. From the resulting Lissajous pattern, the speed may be determined. When the film comes off, the rotor always is slightly disturbed, and this is easily observed by the change in pattern on the screen. Also this rotor disturbance varies the Q of the pickup circuit, which in turn changes the frequency of oscillation of

[10] P. Jacquet, Compt. rend., **118**, 921 (1933).

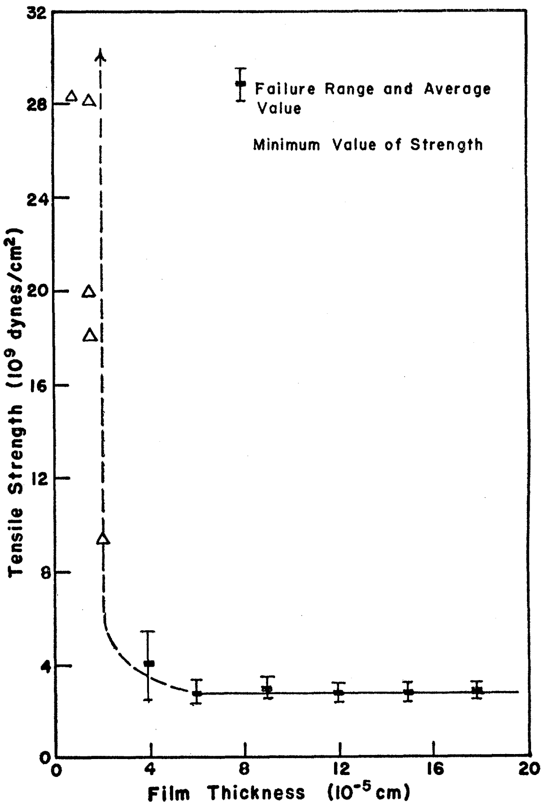

FIG. 3. Curve of tensile strength *versus* film thickness for silver at maximum temperature of 170°K.

the support circuit. Since this frequency is radiated, it may be picked up by a radio receiver in its vicinity. A highly selective RCA type CRV-46131 receiver has been used in this work. With the receiver beat-frequency oscillator in operation, the receiver is tuned to the support-circuit frequency and then detuned to give a low audio-frequency note in the loudspeaker. This note changes pitch abruptly when the film is thrown off the rotor. Also, such a system is useful in monitoring the support circuit.

The film thicknesses were measured by the usual weighing technique, and by light interference methods. In most of the experiments, curves of film thickness *versus* charge transferred in the electroplating process were made. The film thicknesses could then be determined from the amount of charge transferred, with an over-all precision of about one percent.

As pointed out above, the measurement of tensile strength is greatly simplified if the adhesion can be reduced to a negligible value. Consequently, silver films of different thicknesses were deposited on the rotors in patches, and the absolute value of the adhesion A determined by the relation $A=4\pi^2N^2Rhd$. By this procedure the technique of electroplating, described above, for effectively eliminating adhesion was developed and tested. At first sight, it may appear that the patches might loosen around the edges, and that they would "rip off." However, close examination showed this not to be the case. Actually, because of the masking plastic, the deposited films were slightly thinner on the edges, which probably prevents ripping. Also, all of the patches on the rotor came off at approximately the same rotor speed. Incidentally, this method of measuring adhesion has turned out to be very reliable, and gives absolute values.

Figure 3 shows the values obtained for the tensile strength of silver as a function of film thickness. The temperature was above room temperature, the plating current 8.5 ma/cm^2, and the rotor diameter 0.28 cm. It will be observed that for thicknesses greater then 6×10^{-5} cm the tensile strength is about 1.2×10^9 dynes/cm^2, and is independent of the thickness. This is approximately equal to the bulk strength of the silver at the operating temperature. Data not plotted in the figure showed the tensile strength to be independent of film thickness up to at least 10^{-4} cm. For thicknesses between 6×10^{-5} cm and 2.7×10^{-5} cm the data are more erratic, but there is some indication of a small increase in tensile strength. However, for thicknesses less than 2.5×10^{-5} cm, there is a many-fold increase in tensile strength. In fact, the tensile strengths of the thin silver films are so great that the solid steel rotors explode before the films burst. The triangles in Fig. 3

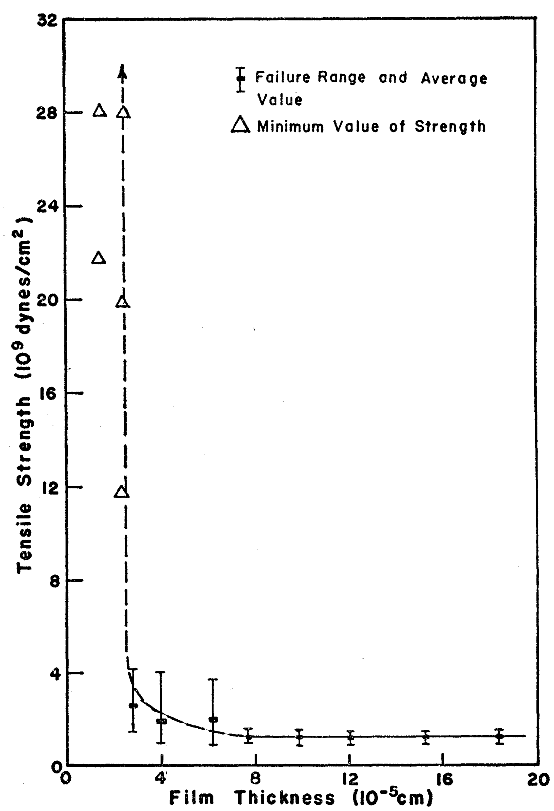

FIG. 4. Curve of tensile strength *versus* film thickness for silver at approximately 330°K.

and Fig. 4 represent points where the rotor speed was not sufficient to burst the film. In some of these cases the rotor was carefully brought to rest and a small cut made across the film in such a way as to eliminate the hoop stress. When the rotors were accelerated again, the films were observed to fly off completely at comparatively low rotor speeds, which was additional evidence that adhesion played no part in the observed tensile strength.

Figure 4 shows a typical plot of film thickness *versus* tensile strength for silver, when liquid nitrogen was introduced into the Dewar flasks surrounding the rotor chamber. The electroplating current was 8.5 ma/cm^2 and the rotor 0.28 cm in diameter. Here again it will be observed that the tensile strength is within experimental error, independent of the film thickness for thicknesses greater than about 6×10^{-5} cm. However, the strengths of these films are somewhat larger (about 2.9×10^9 dynes/cm^2). It is believed that this is the bulk strength of the silver at the temperature of the rotor. Also, as in Fig. 3, the tensile strength increases many times for thicknesses less than 2.5×10^{-5} cm. Because of the rotor heating discussed above, produced by the eddy currents induced in the rotor by the acceleration, the exact temperatures of the rotors used in obtaining the data for both Fig. 3 and Fig. 4 can only be roughly estimated. However, the temperature of the rotor in Fig. 3 was probably around 330°K and that of Fig. 4 at least 160°K lower or a maximum of 170°K. In view of this, the data indicate that the thickness below which the tensile strength shows the very rapid increase is within experimental error, independent of temperature.

First it should be emphasized that the observed strength of the thick films (1.2×10^9 dynes/cm^2) is of the order of 200 times the yield stress of 5.9×10^6 dynes/cm^2 ($3\times10^{-5}\mu$, where μ is the minimum shear modulus) of an annealed single crystal of silver.[4] This may be due to the fact that every grain deforms and hardens by ordinary multiple slip. However, it is possible that the high strength results from the fact that only a fraction of the grains (the larger ones) contain Frank-Read sources[11,12] of length smaller than the grain size (say 10^{-4} or smaller). The piling up of dislocations produced by these sources at the grain boundaries, necessary to nucleate dislocations in the neighboring grains, would then provide the high strength. This interpretation implies, of course, a further increase of strength if the grain size is further decreased. If this interpretation is correct, then it is probable that when the films consist of a monolayer of grains, none of them should be able to stabilize Frank-Read sources in their interior,[13] and therefore the films should quite suddenly increase their strength to the theoretical shear strength. In fact, if an orientation factor of the order of 2 is taken into consideration, the maximum shear strength observed with our thinner films approach within roughly an order of magnitude of the theoretical value.[11,12]

If the greatly increased tensile strength at small film thicknesses is due to the absence of dislocations, the difficulty of generating new dislocations, etc., then one should observe no appreciable slip in the stretched films. However, it is difficult to decide this point, even by the electron microscope, because of the small thickness of the films, but we have in progress special experiments which should not only determine the slip, but also the Young's modulus in films formed both by electrodeposition and by evaporation. Recently, Mr. W. K. Ford and one of us, have found an increase in the tensile strength of evaporated films when the films are very thin, by measuring the bulge of the films through a support, produced by air pressure. However, the results so far, are preliminary, and much less precise than the data of Figs. 3 and 4.

ACKNOWLEDGMENTS

We are much indebted to Dr. H. S. Morton, Jr., and Dr. E. F. Turner, Jr., who carried through a large amount of the preliminary work of this problem. Also, we wish to gratefully acknowledge the help of Professor Nicolas Cabrera for assistance with the theoretical interpretation of the results.

[11] F. C. Frank and W. T. Read, Jr., Phys. Rev. **79**, 722 (1950).
[12] A. H. Cottrell, *Dislocations and Plastic Flow in Crystals* (Oxford University Press, New York, 1953).
[13] W. T. Read, Jr., *Dislocation in Crystals* (McGraw-Hill Book Company, Inc., New York, 1953).